

*D. Leyden*

PHILIP MORRIS U. S. A.  
INTER-OFFICE CORRESPONDENCE  
Richmond, Virginia

To: Dr. Donald Leyden  
From: Harvey Grubbs  
Subject: 1993 Operational Plans for Smoke Research/Technology  
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Objective/Mission Statement

Identify the critical factors necessary to support the development of new products through the measurement and characterization of cigarette smoke and its relationship to cigarette design and consumer acceptance.

Background

This program is being structured to have the capability of characterizing the mainstream and sidestream smoke of any type of cigarette product. Characterization includes determining the analytical, physical, and subjective properties of smoke. All of the appropriate data is to be centralized in a database which will provide a complete record of the design and performance of each sample. The database is the cornerstone of a knowledge base to be built upon the relationships between design and performance characteristics. The value of this program will be its ability to answer the questions of how consumer, manufacturing, regulatory, quality and cost issues affect the products we produce.

The vision for this program is an ambitious one. Ultimately, it will provide a vehicle for evaluating the performance of any cigarette design. This can only be accomplished through the systematic accumulation of a body of data and the reduction of this data to a body of knowledge. But, to provide an immediate resource, the initial strategies of the program are directed toward supporting the existing R&D programs. Through this support, the accumulation of information required will begin with contributions to the more immediate needs of our Company.

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Based on this concept, the following strategies are proposed for 1993.

- I. Smoke generation/collection/analysis/characterization
- II. Internal database/knowledge base and interpretation
- III. Dedicated subjective capability
- IV. Sidestream odor/irritation/visibility
- V. Tomorrow
- VI. Novel smoking articles
- VII. Nicotine delivery/transfer/high taste/low tar

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## Strategy I. SMOKE GENERATION/COLLECTION/ANALYTICAL/CHARACTERIZATION

Rationale: A variety of smoke generation and collection methodologies, and analytical techniques have been previously utilized for smoke characterization. Development of real-time smoke analysis techniques will provide for correlation of measured physical and chemical properties with subjective evaluations of the smoke. Integration of the various analytical techniques into a single structural characterization laboratory would similarly result in more efficient and effective smoke evaluation.

Generation of MS smoke from many types of cigarettes is readily accomplished at the present as is generation of SS for visibility measurement and measurement of SS TPM, CO, CO<sub>2</sub> and hydrocarbons ("CORESTA data"). Methods necessary for labelled tracer studies (total recovery smoking machines) are also available. Analytical techniques for the qualitative evaluation for gas phase, organic gas phase and particulate material by GC with nitrogen, sulfur and oxygen selective detection (oxygen detectors due online by the end of 1992), FTIR and GC/MS have been developed. Literature reports of sidestream studies indicate that various analytical methods are amenable to application as real-time detectors for MS smoke.

Strategy Number 1. Generate, collect, analyze, and characterize mainstream (MS) and sidestream smoke (SS) from any smoking article; ensure that the appropriate methods for such activities are developed and available.

### Tactics and Timetable

#### 1. Smoke Collection/Generation

- a. Refinement of MS smoke generation methods for novel smoking articles  
1 qtr. 93
- b. Improved methods for generation and collection of SS smoke for odor/irritation studies  
2 qtr. 93
- NOTE: This research must be closely coordinated with the development of the dedicated subjective capability
- c. Appropriate delivery and visual measurements of MS with altered smoke presentation/deposition patterns  
3 qtr. 93

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## 2. Smoke Analysis

- a. Initiate development of real-time smoke generation/analysis capability (no pretrapping devices)
  - i. Evaluate atmospheric pressure ionization mass spectrometry, ion mobility spectrometry, particle beam mass spectrometry, direct online GC sample introduction 4 qtr. 93
  - ii. Determine if need exists for real-time smoke analysis by techniques other than FTIR and mass spectrometry (i.e., uv spectroscopy, fluorescence spectroscopy, refractive index) 4 qtr. 93
- b. Develop highly sensitive capability for determination of chemical composition of fresh smoke on a single puff basis 3 qtr. 93
- c. Evaluate the extraction efficiency of supercritical fluid extraction for polar smoke components for automation of TPM fractionation method 2 qtr. 93
- d. Develop methods for measuring the physical properties of the smoke aerosol
  - i. Initiate study of the relationship of particle size and chemical composition. Determine most sensitive size separation methods compatible with qualitative evaluations of the particles 4 qtr. 93
  - ii. Conduct literature study to determine state-of-the-art techniques for single particle analysis 4 qtr. 93
- e. Develop appropriate measurements of time-temperature-space profile of burning coal in a cigarette 4 qtr. 93

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## Strategy II. INTERNAL DATABASE/KNOWLEDGE BASE AND INTERPRETATION

Rationale: Develop databases which will provide (directly or by software linkage) detailed cigarette construction parameters, results of physical and chemical evaluation, results of subjective evaluation and references for experimental details for all samples analyzed.

Existing databases (Semiworks) and database construction already underway for other groups in R&D (CTSD) will be in part applicable to the needs of the Smoke Research Group. Also, there are other PM U.S.A. databases (TLA) which may be accessible for information about tobaccos utilized in model cigarette construction.

Strategy: Evaluate the effects of the cigarette design on the analytical, physical, and subjective character of smoke.

### Tactics and Timetable

1. Design Smoke Research Group database. 1 qtr. 93
2. Create the Smoke Research Group database.
  - a. Links to existing databases 2 qtr. 93
  - b. Completed 3 qtr. 93
3. Develop relationships and correlations among novel filters, tobacco rod density, paper permeability, or banded wrappers and smoke characteristics 4 qtr. 93
4. Provide Cigarette Design Project with knowledge base for modelling applications 4 qtr. 93

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### Strategy III. DEDICATED SUBJECTIVE CAPABILITY

Rationale: A dedicated subjective capability is a requirement for success in the goals of the Smoke Technology program. Analytical measurements are meaningless without the ability to correlate them to subjective evaluations on identical cigarettes. Cigarettes must be evaluated which may not have product potential to obtain the necessary changes in smoke composition to assure a valid correlation.

Strategy: Establish a subjective capability within the Smoke Technology program to permit the subjective evaluation of cigarettes undergoing analytical testing.

#### Tactics and Timetable:

1. Along with PED, establish subjective descriptors suitable for planned studies on SS and MS smoke. 1 qtr 93
2. PED personnel to train subjective panel members drawn from within the Smoke group. 2 qtr 93
3. Utilize and continue to improve the subjective capability so that subjective evaluations may be compared to analytical results in a timely manner, and using smoking articles designed for the investigations. Ongoing

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## Strategy IV. SIDESTREAM ODOR/IRRITATION/VISIBILITY

Rationale: The sidestream smoke strategy is divided into three separate areas to address issues of sidestream irritation, odor and visibility. The strategy for visibility primarily consists of continuing support for the Paper Technology Program. The plans for irritation and odor define the needs for the acquisition of fundamental knowledge necessary to move forward with new product development. The three programs appear on the surface to be distinct and to separately pursue their own objectives. However, it is important to stress the interdependency of each to the others in order to meet the overall objective. There should be frequent interaction between all activities including subjective, analytical and cigarette design to share data and information. For example, a low sidestream cigarette model with a new paper can be evaluated not only for its effect on visibility reduction, but also for its effect on irritation and odor. This kind of interaction would allow assessment of the possibility for development of a product with the combined attributes of reduced irritation, low odor and decreased visibility.

### A. SIDESTREAM ODOR

**Strategy Number 1.** Develop a subjective definition (or subjective descriptors) of the meaning of sidestream odor to the consumer.

#### Tactics and Timetable

1. Conduct a review of the technical literature, PED consumer surveys and in-house data to ascertain the current state of knowledge as it relates to sidestream odor.  
1 qtr. 93
2. Define the subjective differences between fresh and aged sidestream smoke to establish the time scale over which the change of odor occurs and explore the effects of oxygen and pH on the subjective characteristics of sidestream smoke.  
3 qtr. 93
3. Define subjectively the meaning of room or lingering odor and investigate the effects of sidestream smoke on room characteristics.  
2 qtr. 93
4. Establish if a differentiation, if any, exists between ashtray odor and room (lingering) odor.  
2 qtr. 93
5. Evaluate the roles of the gas and particulate phases of sidestream smoke as they relate to their contribution to sidestream smoke odor.  
4 qtr. 93

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6. Define conditions necessary for consistent odor generation to facilitate the analytical study. 1 qtr. 93
7. Review and relate to the odor issues any data generated in the sidestream irritation plan. 4 qtr. 93 and continuing

Strategy Number 2: Develop analytical methodology integrated with subjective odor evaluation to attempt to identify compounds that are significant contributors to sidestream smoke odor.

Tactics and Timetable

1. Devise suitable sampling methods to collect the matrix containing odor characteristic compounds. 1 qtr. 93
2. Design a fractionation protocol, guided by odor evaluations, to locate those fractions most likely to represent the sidestream odor. 3 qtr. 93
3. Use multidimensional gc/ms in conjunction with organoleptic evaluations to analyze the odor contributing fractions from 2.2. Rank the components in each fraction by their odor threshold and identify those with the lowest threshold. 4 qtr. 93
4. Conduct a dose response study based on the results determined in 2.3. 4 qtr. 93

Strategy Number 3: Design methodologies to assess the feasibility of reducing and/or masking the chemical compounds determined to be responsible for sidestream smoke odor.

Tactics and Timetable

1. Review the methodologies used in the sidestream smoke irritation operational plan and assess the effects of these methodologies on addressing the issue of sidestream odor-contributing compounds. 4 qtr. 93 and continuing

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## B. SIDESTREAM IRRITATION

Strategy Number 1: Develop subjective assessment tools to measure eye and nose irritation to the smoker and/or non-smoker from the fresh, concentrated sidestream plume.

### Tactics and Timetable

1. Develop a reproducible test to measure sidestream irritation as it relates to the smoker. 1 qtr. 93
2. Review and evaluate the current state of sensor technology with particular emphasis on the capability to "sense" those compounds in cigarette smoke reported to be irritants. 2 qtr. 93
3. Initiate studies to evaluate the potential use of gas sensors as tools in the study of the irritation of sidestream smoke. 4 qtr. 93
4. Utilize a panel "booth" with controlled conditions of temperature, humidity and air circulation to assess sidestream smoke irritation to the smoker. 2 qtr. 93
5. Determine the optimum method for the generation of fresh sidestream smoke for sensory evaluation. 1 qtr. 93
6. Determine the feasibility of constructing an in-house facility, i.e., eye and nose "sniff-port", to study sensory properties of individual sidestream smoke components. 2 qtr. 93
7. Generate and analyze specific analytical data in relation to information obtained from the previously conducted literature search on known irritants and from external expertise. 2 qtr. 93
8. Evaluate the relevance of literature and analytical data to the actual irritation of sidestream smoke, i.e., is there a group of compounds present above threshold levels or a large number of compounds, none of which are estimated to be present above threshold. Are these materials present in the gas or particulate phases and how are they transported to the subject? 4 qtr. 93
9. Determine the need to design and construct an appropriate facility or identify an appropriate facility to conduct sidestream smoke sensory studies.

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1 qtr. 93

10. Utilizing information from the literature and the above studies, generate a list of hypotheses to test the importance of various smoke components and cigarette properties to irritation. Continuing
11. Develop approved methodology for addition of model compounds or select portions, i.e., gas phase of sidestream smoke. 4 qtr. 93
12. Evaluate dose response (concentration effects) to estimate threshold levels of irritants and/or combinations of irritants. 1994
13. Compare the threshold levels of specific compounds against analytically determined quantities for those compounds to arrive at their relative contribution to the overall irritation of smoke. Also, decreasing amounts of smoke may be used directly or after chromatography until threshold levels are reached. 1994

**Strategy Number 2:** Develop analytical methodology to determine compositional differences between selected test cigarettes and controls and relate the differences; if possible, to known classes of irritants.

**Tactics and Timetable**

1. Design and construct a direct inlet system interface to a CI mass spectrometer to facilitate the instantaneous mapping of the molecular weight distribution of the sidestream smoke plume. 1 qtr. 93
2. Devise and utilize methodology to measure the particle size and determine the thermal properties of the sidestream smoke plume. Construct "profiles" of the sidestream smoke plume to allow comparisons between models of varied construction to attempt to define differences that may relate to sidestream smoke changes. 1 qtr. 93
3. Develop methodology to determine the composition of sidestream gas phase for compounds containing nitrogen, sulfur or oxygen by gas chromatography/mass spectrometry. 3 qtr. 93 continuing
  - a. Simultaneously analyze sidestream smoke from selected cigarette models for sulfur and nitrogen containing compounds using a Sulfur Chemiluminescence Detector (SCD) in tandem with a Nitrogen-Phosphorus Detector (NPD). Total analysis capability

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requires data collection, data reduction, statistical evaluation and compound identification.

- b. Analyze sidestream gas phase for oxygen-containing compounds, including ketones, aldehydes, acids and alcohols by an Oxygen-selective Flame Ionization Detector (O-FID) and/or by the use of an Atomic Emission Detector (AED). An AED would permit the analysis of oxygen containing materials as well as compounds containing a number of other elements.
  - i. This requires the evaluation of the performance of O-FID at the vendor's laboratory using smoke extracts. This has been done but results have not been obtained. If the results are acceptable, the purchase of this detector will be recommended.
  - ii. Conduct smoke analyses of model cigarettes, data collection, analysis and interpretation.
4. Determine the effect of inorganics in paper (including fillers and fluxing agents) on sidestream smoke chemical compositions and sensory properties by evaluating low sidestream models made with wrappers containing these fillers. An AED could be of use here in determining the transfer of inorganics to smoke.

2 qtr. and continuing
5. Determine the effects of the thermal properties of the smoke plume on the chemical and sensory attributes of the sidestream smoke.

2 qtr. 93
6. Develop methodology to assess the gas phase/particulate partitioning of compounds that are shown to be irritants in sidestream smoke. Utilize this methodology to determine the effects of temperature and dilution on the delivery mechanisms of the irritants to the subject.

4 qtr. and continuing

Strategy Number 3: Assess possible methods of reducing the levels of those chemical classes in sidestream smoke which are most likely to contribute to irritation.

#### Tactics and Timetable

1. Design and construct a reactor to evaluate thermal and catalytic cracking of sidestream smoke.

2 qtr. 93

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2. Develop model systems to study interactions of specific classes of compounds with selected inorganic materials. 4 qtr. 93
  - a. Conduct theoretical modeling of residence time and temperature to determine the required kinetic parameters for a catalysis process. 2 qtr. 93
  - b. Conduct a literature search to identify approved materials with reported catalytic properties for destruction of the classes of compounds that are most likely to contribute to irritation. 2 qtr. 93
  - c. Evaluate low sidestream fillers, fluxing agents and interaction products of these with each other and with cellulose, e.g., char, for catalytic activity with model compounds representing chemical classes related to irritation. 4 qtr. 93
3. Develop methods to evaluate cigarettes sensorially for irritation and analytically for compositional differences. 3 qtr. 93
4. Prepare low sidestream cigarettes with magnesite, hydromagnesite/brucite and calcium carbonate fillers at optimum reduced visibility levels and evaluate these sensorially for irritation and analytically for compositional differences. 4 qtr. 93
5. Conduct imaging studies on cigarettes incorporating magnesite, hydromagnesite/brucite and calcium carbonate in the wrapper. 4 qtr. 93
6. Identify inorganic materials with potential for catalysis, prepare candidates and evaluate the effect of physical properties, i.e., morphology, particle size, surface area, as well as, chemical and thermal properties on catalytic function. 1994
7. Using model compounds and selected smoke fractions, evaluate catalytic and thermal effects of candidate catalysts under conditions extant in the burning cigarette and characterize products both analytically and sensorially. 1994
8. Evaluate promising catalytic materials in cigarette models subjectively and analytically. 1994

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9. Develop appropriate facilities, methods and protocols to conduct sensory evaluations of products from thermal cracking studies. 1994
10. Evaluate blend additives or changes in blend composition which will reduce irritation while maintaining subjective acceptability. 1995

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### C. SIDESTREAM VISIBILITY

Strategy Number 1. Provide assistance as required in the design, construction and operation of instruments to determine the quantities of sidestream smoke from conventional and low sidestream models.

#### Tactics and Timetable

1. Provide the necessary information for the construction of an 8-port visibility monitor at the R&D Facility in Neuchatel, Switzerland. Assist in the construction and instrument check-out as requested. 1 qtr. 93
2. Determine the relationship of sidestream visibility reduction to changes in mainstream subjectives and to changes in sidestream odor and irritation. The appropriate operational plans should be reviewed for addressing this tactic. 1 qtr. and continuing
3. Provide assistance and support to those areas currently using sidestream visibility instruments. Design and construct additional instruments as requested. continuing

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## Strategy V. TOMORROW

Rationale: Project Tomorrow has the responsibility of ensuring that all of our domestic cigarettes pass average ignition-propensity standards which may be legislated. This may require our cigarettes to be substantially re-designed in order to meet such standards.

Strategy: Support project Tomorrow in the development of new cigarette designs.

### Tactics and Timetable

1. Assess the impact of cigarette design parameters on the deliveries and subjective character.

a. Tobacco rod densities	3 qtr. 93
b. Cigarette permeability	3 qtr. 93
c. Banded wrappers	4 qtr. 93

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## Strategy VI. NOVEL SMOKING ARTICLES

Rationale: The immediate need of the program is the ability to characterize the composition of the aerosol and relate it to both the design/control parameters and subjective response. Characterization of the aerosol can consist of a detailed description of the chemical makeup as conventionally done, and/or simply a molecular weight distribution, which has not been done before. This new methodology may also be applicable to the characterization of cigarette smoke which is known to contain macromolecular species (>100,000 Daltons).

A significant difference between the aerosol generated at low temperature and the cigarette smoke is the water content. With a similar nicotine/tar ratio, the nicotine impact and overall response can be significantly higher than expected from the FTC tar delivery. This plan incorporates steps to address the effect of water.

Strategy: Establish the relationships among tobacco thermal history, aerosol composition and the subjective quality of the aerosol generated at low temperature.

### Tactics and Timetable

1. Conduct compositional analyses of the aerosol generated at low temperature and relate composition to the thermal history of the aerosol generation matrix.
  - a. Use currently available methods to define the chemical composition of the aerosol.

1 qtr. 93
  - b. Develop methodology for a more complete and rapid single-puff analysis of the aerosol.

3 qtr. 93
2. Determine the relationship between aerosol composition and subjective response for underheated, properly heated and overheated aerosol generation matrix.
  - a. Assess the feasibility of using the molecular weight distribution, particle size distribution or acid/base property of the aerosol as a simple and reliable predictor of subjective quality.

3 qtr. 93
  - b. Evaluate the effect of water on the subjective response and the possible synergistic effect with the tar and nicotine delivery.

3 qtr. 93

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- c. Separately determine the importance of the volatile distillates and pyrolyzates to subjective response in order to optimize the heating conditions needed to generate subjectively acceptable but chemically simple aerosol.

4 qtr. 93

- d. Consult with High Taste/Low Tar program for possible inclusion of its findings in prototype optimization.

Continuing

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## Strategy VII. NICOTINE DELIVERY/TRANSFER; HIGH TASTE, LOW TAR

Rationale: The ability to design cigarettes which are low in tar but rich in flavor and satisfaction has long been a goal of R&D. Systematic investigations aimed toward a practical end point will provide cigarette designers the information needed to accomplish this.

Strategy: Evaluate the effects of cigarette design on the impact of low delivery products. Specifically, determine the effects of blend composition, tobacco and filter additives and cigarette designs on the subjective impact of smoke and the ratio of nicotine to tar.

### Tactics and Timetable

1. Review past studies involving filler and filter additives and novel cigarette and filter constructions.  
1 qtr. 93
2. Work with the Leaf Department and the Tobacco Technology Group to design blends which have high nicotine-to-tar ratios and incorporate them into cigarettes designed to deliver five milligrams of tar, or less; evaluate the smoke and assess the concept.  
1 qtr. 93
3. Work with the Flavor Technology Group to design filler and filter additives which enhance the impact of smoke; incorporate them into ultra-low delivery products and evaluate their smoke.  
1 qtr. 93
4. Work with the Filter Technology Group to assess the influence of novel filter materials and constructions on the impact of the smoke from ultra-low delivery products.  
4 qtr. 93

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## Resources

The following personnel and/or expertise would be required to initiate a Smoke Technology/Research Program. Other expertise would be added as the program progressed.

GC, GC/MS, pyrolysis GC/MS	4
HPLC	1
Spectroscopy	1
Aerosol Chemist	2
Radiochemist	2
Computer-Data input	0.5
Data base manager	1
Catalysis Chemist	1
Organic Chemist	1
Inorganic Chemist	1
Subjective panel coordinator	1
Technician	3
Cigarette designer	0.5
Blend designer	0.2
Flavor specialist	0.2
Filter designer	0.2
Panelists (some (all) from members of Smoke Technology)	10

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